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Performance (Technical) Report



March 1, 1993 to February 28, 1994

"Tunneling and Transport in Mesoscopic Structures"



OFFICE OF NAVAL RESEARCH

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## **Summary**

This program is focused on developing processes for the fabrication of arrays of side-byside tunneling junctions and single side-by-side junctions. The approach combines the use of electron beam lithography, ion milling, and reactive ion etching to produce structures on Si wafers which can serve as contact shadow masks for low-temperature, with an angled vapor deposition step which is used to complete the array by forming junctions. We have produced shadow masks with 300 Å-wide channels at the National Nanofabrication Facility at Cornell. We have configured our deposition system so as to achieve precision alignment of the direction of the vapor stream relative to the array structure, and are proceeding with the fabrication and electrical characterization of arrays. Electron microscope studies indicate that we have achieved an acceptable level of alignment for the purposes of the project, and low temperature depositions are under way at this writing. The structures being fabricated will involve junction feature sizes smaller than those produced to date using other processes. The flexibility of the process will permit the fabrication of arrays with various levels of disorder thus facilitating the investigation of the effects of disorder on the superconducting and normal state properties of the structures as well as on recently predicted quantum Hall effect behavior in such structures. The metalization currently being used involves the deposition of Pd from a wire-fed electron beam evaporation source. By hydrogenating the Pd electrodes their ordinary metallic character can be changed such that they become superconducting with transition temperatures of the order of 8 K. This possibility will permit the comparison of behavior in the normal and superconducting states of the same structures while avoiding complications associated with the application of magnetic fields which are used to extinguish superconductivity in most of the structures of this type being studied.

The most significant achievement of this program to date has been the successful fabrication of metallic wires and wire arrays with widths the order of 50-150 Å. To our knowledge these are the narrowest wires fabricated anywhere in the world. These structures were produced in failed attempts to produce junctions as a consequence of misalignment of the evaporation source with the axes of the shadow mask and the plane of the substrate. Although the program is focused on the study of junction structures, these wires, which are produced using a slight variant of the angled evaporation process, should provide an additional and unexpected opportunity for the study of small scale physical phenomena.

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#### 1.0. Introduction

This research effort is directed at the study of specially prepared arrays of tunneling junctions which will have feature sizes significantly smaller than those produced using conventional state-of-the-art lithographic techniques. The fabrication procedure is such that either positional or junction coupling disorder can be introduced into the arrays in a controlled manner. The work is motivated in part by the need to produce physical systems which are close in their geometries to physical models of the superconductor-insulator transition in ultrathin films (Fisher, 1990, and Cha et al., 1991). The underlying physical model in these systems is a Josephson junction array which is dominated by the site-specific electrostatic energy. This is in contrast with other models of junction structures in which the dominant electrostatic energy is the inter-site energy (Fazio and Schon, 1991). In real arrays both energies play a role, and it would be desirable to alter the relative strengths of the two contributions to the electrostatic energy, as a means of validating the models. The advantage of the present approach over earlier ones is the possibility of producing artificial structures which realize important features of real films, as well as coming very close to constraints of theoretical models. An important benefit of this project is that it will open up for investigation a regime of mesoscopic device structures with feature sizes smaller than those currently under investigation at other laboratories around the world.

The research is being carried out using a unique combination of what might be termed "conventional" microstructure fabrication techniques such as direct-writing electron beam lithography, lift-off, and reactive ion etching, with a clearly unconventional approach to metalization, in situ deposition of metal onto substrates held at low temperatures (Orr and Goldman, 1985). The first step is to produce a contact "shadow" mask on a substrate using nanometer scale lithography. This mask, which is permanently affixed to the substrate, ultimately will be a part of the side-by-side tunneling structure, or array of junctions. The key to the process is to narrow the gaps or channels separating metallic mesas on the substrate which are in effect the mask, so as to form the sought after side-by-side tunneling junctions or junction arrays. The crucial feature of the procedure is the suppression of surface diffusion of the evaporant by carrying out the deposition with the substrate cooled to liquid helium temperatures. If this process were to be carried out at room temperature it would not be possible to achieve delicate control of the coupling of the mesas because of diffusion. The other feature of the process is the capability of monitoring the formation of the structures in real time through the measurement of the resistance and capacitance of the array, or of single test junctions, during the deposition process.

This process will inherently be a flexible one in permitting the variation of designated properties of the arrays. By changing the deposition angle, or the amount of material deposited, the Josephson coupling energy can be altered. Through modification of the software used to generate the shadow mask, the site-occupation probability and the channel width can be altered, thus permitting the systematic alteration of the disorder. Decisions at the time of the fabrication of the shadow mask can control the Coulomb energy. The possibility of hydrogenating the electrodes can permit a normal metal to be converted to a superconductor at low temperature (Buckel, 1979, Skoskiewicz, 1972, and Stritzker and Buckel, 1972).

The program of scientific investigations will initially be concerned with studies of transitions between normal, superconducting and insulating states as a function of temperature (Fisher, 1990, Haviland et al., 1989, and Cha et al., 1991). We will study the effect on these transitions of the Coulomb interaction which depends on the ration of the inter-island or junction capacitance, C, and the self capacitance of the islands, C<sub>0</sub>. This ratio as mentioned above can be varied by adjusting the geometry of the arrays. There are a number of different studies which are planned. These include the investigation of coherent states at low temperatures (Jacobs et al., 1988), the study of the charge Kosterlitz-Thouless transition in these systems under the

conduction  $C/C_0>>1$  with either normal or superconducting electrodes (Mooij et al., 1990). This phenomenon is an analog of transitions involving vortices which have been studied in superconductors and superfluids. Finally, the most amazing opportunity is the possibility that the vortices in an array of junctions in a magnetic field resemble in their properties the electrons a two-dimensional lattice in a magnetic field perpendicular to the lattice. Under certain conditions, this resemblance may result in the formation of a quantum Hall fluid of vortices. There is a possibility that such a quantum Hall fluid of vortices exhibits quantized Hall electronic transport in the manner of the quantum Hall effect (Stern, 1994).

The second general direction will be to introduce disorder systematically. We would search for glass behavior which we have found in wire networks produced using conventional  $0.1 \, \mu m$  lithography. The junction arrays are much closer to the theoretical models. In such disordered arrays we would also investigate the phenomenon of vortex hopping which has a quantum limit which involves vortex tunneling (Fisher, Tokuyasu, and Young, 1991).

### 2.0. Progress

In the previous report we described our progress in the fabrication of the contact shadow masks at the National Nanofabrication Facility at Cornell. These masks consist of arrays of square islands separated by gaps of the smallest widths obtainable. We have characterized the structures using scanning electron microscopy (SEM) and optical microscopy. The edges of the islands in the arrays have nearly square profiles in order for the angle evaporation to coat only specifically intended areas. For completeness we show the current chip design in Fig. 1, and the SEM photographs, Figs. 2, and 3, show a linear test array and its contacts, and the contact for Hall measurements of the 2-D array. As the process for producing arrays was described in detail in the previous report, it will not be discussed here.

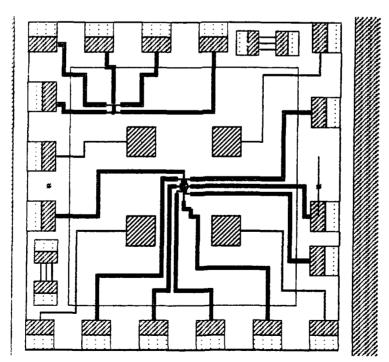
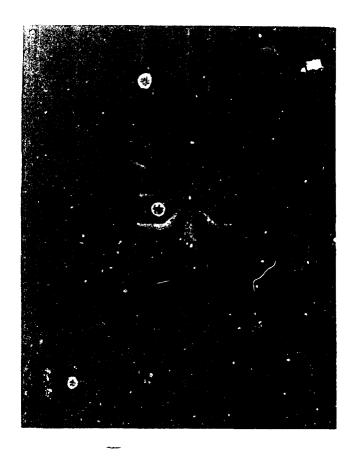


Fig. 1: The chip design. The two-dimensional array is at the center, while the linear array is located toward the upper left. The larger pads around the outside are for electrical contact. The large cross just above center is an electron beam alignment mark. The structure at center left and center right are photolithography alignment marks. Also visible are the capacitive leveling pads, the four large pads near the main array, and the etch test structures, the pairs of smaller pads connected by three parallel lines. The final two types of structures are described in more detail below.



14g. 2: An SEM photograph of the linear array and its contacts.

Work in the current period has been concerned with perfecting the evaporation process. The most important problems which had to be solved were difficulties with the Pd E-beam evaporation system, and in controlling the orientation of the vapor stream relative to the pattern. The first problem necessitated that the evaporator be returned to the manufacturer in Europe, resulting in some delay in the project. The evaporation source is now operating according to specification, and high-quality Pd films are made easily and regularly.

The second problem which also resulted in some delay was in controlling the angle of evaporation, and consequently the alignment of the evaporation. It turned out that as a consequence of differential contraction in the manipulator of the flow through cryostat during cooldown to liquid helium temperatures, the alignment of the source, accomplished at room temperature, was repeatedly lost. The solution to this problem was the development of an alignment scheme operative at liquid helium temperatures. This uses a laser to determine a precise angle, and necessitated the modification of the vacuum system with the incorporation of an additional tilt device. We can now rotate the sample holder, and tilt it about three orthogonal axes.

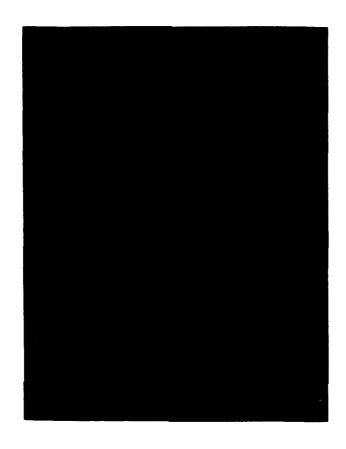


Fig. 3: An SEM photograph showing a contact to a single island. These contacts are for Hall measurements of the 2-D array.

We are currently engaged in running the system, attempting to control the process step in which junctions structures are made. We have confirmed that we can produce the "bow-tie" structure that constitutes the desired junction bridge. The most surprising result was that misaligned evaporations result in structures which may be useful in their own right. When the angle of the vapor stream with respect to the array axes and the substrate plane is such that the shadowing is not complete, it is possible to produce extraordinarily narrow wires. We have measured their widths to be between 50 and 150 Å, depending upon the particular run. To our knowledge, these wires are the narrowest made anywhere in the world. They have smaller cross-sections than the structures produced by Sharifi et al. (1991). A field emission and convention scanning electron microscope photographs of parts of a narrow wire array are shown in Figs. 4 and 5. These structures appear to be robust, in that they survive being warmed to room temperature and removed from the evaporation system. An account of the process leading to these structures is being prepared for submission to Applied Physics Letters.

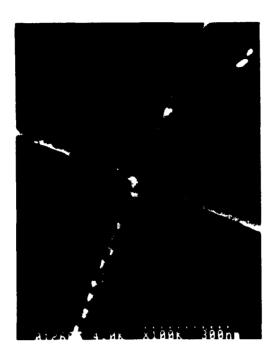


Fig. 4: Backscattered electron image from a field emission SEM showing the corners of four islands and the bow-tie shaped structure that lies in this intersection. The in-plane component of the Pd beam comes from the upper right. This beam was too close to the normal, shorting the triangular junction structures to the neighboring islands. The resistance of this array was approximately 4x10<sup>4</sup> ohms. The wires shown in this figure span the entire 80 µm square array of 80x80 cells. The live average widths are 150 Å. We have made arrays which have line widths of less than 100 Å ( ~ 70 Å). These, to our knowledge, are the narrowest wire structures that have been prepared anywhere.



Fig. 5: Split screen image of a similar region in a different array. The upper image is a secondary electron image and the lower is a backscattered electron image. Again, this array was misaligned, shorting adjacent islands together through the junction structures and giving a resistance of the order of 10<sup>4</sup> ohms.

#### 3.0. Future Work

Since we are now performing the angled evaporations on actual arrays, we expect to have useful junction structures in the immediate future. We can reproducibly generate narrow wires. We have determined that electrical resistance measurements are more useful than capacitance measurements for process control, and during evaporations have been carrying out the former. We plan to continue to characterize structures by measuring their capacitances. We expect to begin hydrogenation of junctions and wires in the very near future. We are planning a trip to the National Nanofabrication Facility at Cornell to fabricate disordered arrays as soon as a process run resulting in good junctions is carried out.

The scientific investigations which depend on the technology we are developing should be initiated in the very near future. With the structures we have in hand we should be able to study both the phase transitions in junction arrays between metallic, insulating, and superconducting behaviors as well as the quantum Hall effect analogs mentioned above. In the matter of wires, we have unique opportunities to study superconductivity, localization, and magnetism in the one-dimensional limit.

#### 4.0. Personnel

The personnel working on this project include Dr. Gabriel Spalding, Mr. Eric Olson, and Ms. Gloria Martinez-Arizala. Spalding is paid from project funds, and Olson and Martinez-Arizala are paid half each from University matching funds. The remainder of Olson's support comes from a Department of Education Fellowship, and Martinez-Arizala is partially supported by a University Minority Student stipend.

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## 6.0. Publications and Presentations

An account of this work was presented by Mr. Eric Olson at the 1994 APS March Meeting in Pittsburgh. A copy of the abstract is attached.

"Fabrication of Side-by-Side Junctions Exhibiting Vacuum Tunneling," Eric Olson, G. C. Spalding, Michael Rooks, and A. M. Goldman, Bull. Am. Phys. Soc. 39, 249 (1994).